Naval Research Laboratory

Washington, DC 20375-5320

NRL/MR/6930--17-9736

Bioinspired Surface Treatments for Improved Decontamination: Commercial Products

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July 28, 2017

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*	**2. REPORT TYPE**	**3. DATES COVERED** *(From - To)*
28-07-2017	NRL Memorandum Report	October 7, 2015 – January 30, 2017

4. TITLE AND SUBTITLE
Bioinspired Surface Treatments for Improved Decontamination: Commercial Products

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
Brandy J. White, Anthony P. Malanoski, and Martin H. Moore

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER
69-1C75

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Naval Research Laboratory, Code 6930
4555 Overlook Avenue, SW
Washington, DC 20375-5320

8. PERFORMING ORGANIZATION REPORT NUMBER
NRL/MR/6930--17-9736

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Defense Threat Reduction Agency
DTRA-Joint CBRN Center of Excellence
BLDG E-2800
APG-EA, MD 21010

10. SPONSOR / MONITOR'S ACRONYM(S)
DTRA–CB10125

11. SPONSOR / MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION / AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT
This effort evaluates bioinspired coatings for use in a top-coat type application to identify those technologies that may improve decontamination capabilities for painted surfaces. This report details results for evaluation of four commercially available treatments: NANOskin Hydro Express, Rust-Oleum® NeverWet®, Eagle One Superior NanoWax™, and Rust-Oleum® Wipe New. Commercially available treatments offer some potential advantages over the less mature technologies otherwise evaluated by this effort. Hazards, deposition approaches, and production scaling have been addressed, and the materials are suitable for immediate use as appropriate. Given the potential for rapid adoption and reduced costs, evaluation of relevant commercial treatments was completed following standard approaches. Retention of the simulants paraoxon, methyl salicylate, dimethyl methylphosphate, and diisopropyl fluorophosphates following treatment of contaminated surfaces with a soapy water solution is reported. Wetting behaviors and target droplet diffusion on the surfaces are also discussed.

15. SUBJECT TERMS
Coatings
Paint
Decontamination

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**			Brandy J. White
Unclassified Unlimited	Unclassified Unlimited	Unclassified Unlimited	SAR	29	**19b. TELEPHONE NUMBER** *(include area code)* (202) 404-6100

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. Z39.18

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EXECUTIVE SUMMARY

The Center for Bio/Molecular Science and Engineering at the Naval Research Laboratory (NRL) initiated a program in January 2015 for evaluation of bioinspired treatments suitable for use as a top coat on painted surfaces with the intention of achieving improved aqueous decontamination of these materials. Funding was provided by the Defense Threat Reduction Agency (DTRA, CB10125). As part of this effort engagement with scientists in academia and industry was used to identify technologies that offered potentially useful characteristics. This report details results for evaluation of four commercially available surface treatments: NANOskin Hydro Express, Rust-Oleum® NeverWet®, Eagle One Superior NanoWax™, and Rust-Oleum® Wipe New. Retention of the simulants paraoxon, methyl salicylate, dimethyl methylphosphate, and diisopropyl fluorophosphate following treatment of contaminated surfaces with a soapy water solution is reported along with droplet diffusion on the surfaces and wetting angles.

BIOINSPIRED SURFACE TREATMENTS FOR IMPROVED DECONTAMINATION: COMMERCIAL PRODUCTS

INTRODUCTION

The DoD Chemical and Biological Defense Program (CBDP) seeks to provide protection of forces in a contaminated environment including contamination avoidance, individual protection, collective protection, and decontamination. In January 2015, the Center for Bio/Molecular Science and Engineering at the Naval Research Laboratory (NRL) began an effort funded through the Defense Threat Reduction Agency (DTRA, CB10125) with a view toward evaluation and development of top-coat type treatments suitable for application to painted surfaces that would reduce retention of chemical threat agents following standard decontamination approaches. The effort sought to survey relevant and related areas of research and evaluate identified technologies under appropriate methods to determine efficacy, scalability, and durability.

The current document summarizes results for evaluation of four commercially available surface treatments: NANOskin Hydro Express, Rust-Oleum® NeverWet®, Eagle One Superior NanoWax™, and Rust-Oleum® Wipe New (Figure 1). These surface treatments were identified through interaction with academic and industrial scientists familiar with the technologies and with the goals of this ongoing effort. Commercially available treatments offer some potential advantages over the less mature technologies otherwise evaluated by this effort. Hazards, deposition approaches, and production scaling have been thoroughly addressed. The materials are suitable for immediate evaluation and movement into use as appropriate. Given these factors and the associated potential for rapid adoption and reduced costs, evaluation of relevant commercial treatments was determined to be a valuable exercise. Treated coupons were subjected to standard evaluations including measurement of sessile, sliding, and shedding contact angles and quantification of retention for the simulant compounds.

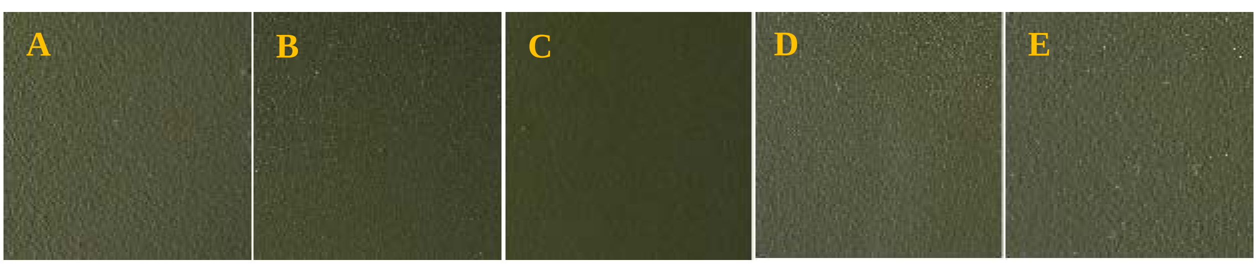

Fig. 1 — Images of a painted coupon (A), a Nanoskin treated coupon (B), a Wipe New treated coupon (C), a NeverWet treated coupon (D), and a NanoWax treated coupon (E).

NANOskin Hydro Express, referred to as Nanoskin here, is a spray polymer treatment intended for application to paint. Advertising claims indicate finish restoration, three month durability, water repellence, static dissipation, and protection of the finished surface. The safety data sheet for the product declares it to be an aqueous solution of octene polymer with ethene, a proprietary conditioner, and ethylene acrylic acid as a copolymer.

Manuscript approved June 12, 2017.

Rust-Oleum® NeverWet® is a spray applied, liquid repelling treatment available in several packaging variants: Hunting & Outdoor, Auto Interior, Boot & Shoe, Fabric, Rain Repellent, and Liquid Repelling Treatment. These products vary in application method and in constituents. Here, we have used the product intended for use on automotive interior surfaces. Advertising claims indicate that the product produces a clear superhydrophobic coating that improves resistance to moisture and stains with no impact on the appearance of the surface. The safety data sheet for the product declares the content to be petroleum lubricating oils (C15 to C30) and ethylene glycol methyl ether. Other formulations identify hydrotreated light distillate, mineral spirits, and aromatic petroleum distillates. The Rain Repellent and multicomponent Liquid Repelling Treatment appear to be distinct from the other products.

Rust-Oleum® Wipe New is available in several packaging variants: Trim Restore, Headlight Restore, ReColor, Wheels, and Tires. The Trim Restore, Headlight Restore, and ReColor packages contain 1-chloro-4-(trifluoromethyl)benzene and benzaldehyde according to the safety data sheets. The Wheels and Tires packages vary slightly from the other products and contain tert-butyl acetate in addition to the other components. Here, we have used the Headlight Restore package. The material is supplied on a preloaded microfiber cloth to be wiped onto the surface. Advertising claims for this group of products include restoring and protecting plastics, trim, vinyl, rubber, metal, and stone.

Eagle One Superior NanoWax™ is a spray applicable formula that incorporates nanoparticles of carnauba wax. The claims associated with this product are repellence of positively charged particles (dust, for example), improved durability, and filling of scratches and damage leading to restoration of the finish. The safety data sheet lists carnauba wax and ethylene glycol as components. This product was selected as a representative of available automotive spray on wax treatments. It is suitable for use on painted, glass, and plastic surfaces.

METHODS

Aluminum coupons were painted with a polyurethane based system following the directions for those products. Deposition of the surface treatments onto painted was completed as advised by manufacturer directions. All drying and curing steps were completed under laboratory ambient temperature and humidity. NANOskin Hydro Express is shaken and sprayed onto a clean, cool surface. The product is spread evenly on the surface and allowed to dry. Eagle One Superior NanoWax™ is similarly applied by spraying and wiping. Rust-Oleum® NeverWet® is applied by simply spraying onto the surface and allowing to dry. Rust-Oleum® Wipe New is provided in a kit with a polishing pad and a preloaded microfiber towel. The polishing pad is used with water to clean old surfaces. Here, we report values for coupons that were not polished; no differences in performance (wetting or retention) were noted for polished coupons. The product is applied by wiping onto the surface.

Sessile contact angles for samples evaluated under this effort used three 3 μL droplets per surface with each droplet measured independently three times for each of three targets, water, ethylene glycol, and n-heptane. Geometric surface energy was calculated based on the water and ethylene glycol interactions using software designed for the DROPimage goniometer package. Sliding angles were determined using 5 μL droplets. The droplet was applied at 0° after which the supporting platform angle was gradually increased up to 60°. Sliding angles for each of the liquids were identified as the angle for which movement of the droplet was identified. Shedding angles for each liquid were determined using 12 μL droplets initiated 2.5 cm above the coupon surface. Changes in base angle of 10° were utilized to identify the range of droplet shedding angle based on a complete lack of droplet retention by the surface (not sliding). The angle was then reduced in steps of 1° to identify the minimum required angle. Droplet diameters were determined using tools provided by Adobe Photoshop CS3. Droplets of 5 mL were applied to the surfaces and images were collected at 30 s intervals for 5 min followed by images at 5 min intervals for a total of 30

min. DFP samples were kept covered for the duration of the experiment to minimize evaporation. In some cases, reflections from the glass cover can be seen in the images.

Simulant exposure and evaluation methods were based on the tests developed by Edgewood Chemical Biological Center referred to as Chemical Agent Resistance Method (CARM). [1] Standard target exposures utilized a challenge level of 10 g/m^2; work completed under studies leading to the CARM protocol indicate that retention scales with challenge level. Here, the coupons were 0.00258 m^2; a 5 g/m^2 target challenge was applied to the surfaces as a two equally sized neat droplets. Following application of the target, coupons were aged 1 h prior. Decontamination used a gentle stream of air to expel target from the surface prior to rising with soapy water (0.59 g/L Alconox in deionized water). The coupons were then soaked in isopropanol for 30 min to extract remaining target; this isopropanol extract was analyzed by the appropriate chromatography method to determine target retention on the surface.

For paraoxon analysis, a Shimadzu High Performance Liquid Chromatography (HPLC) system with dual-plunger parallel flow solvent delivery modules (LC-20AD) and an auto-sampler (SIL-20AC; 40 μL injection volume) coupled to a photodiode array detector (SPD-M20A; 277 nm) was used. The stationary phase was a C18 stainless steel analytical column (Luna, 150 mm x 4.6 mm, 3 μm diameter; Phenomenex, Torrance, CA) with an isocratic 45:55 acetonitrile: 1% aqueous acetic acid mobile phase (1.2 mL/min). [2] For analysis of methyl salicylate (MES), diisopropyl fluorophosphate (DFP), and dimethyl methylphosphonate (DMMP), gas chromatography-mass spectrometry (GC-MS) was accomplished using a Shimadzu GCMS-QP2010 with AOC-20 auto-injector equipped with a Restex Rtx-5 (30 m x 0.25 mm ID x 0.25 μm df) cross bond 5% diphenyl 95% dimethyl polysiloxane column. A GC injection temperature of 200°C was used with a 1:1 split ratio at a flow rate of 3.6 mL/min at 69.4 kPa. The oven gradient ramped from 50°C (1 min hold time) to 180°C at 15°C/min and then to 300°C at 20°C/min where it was held for 5 min.

RESULTS

Each of the commercial products was applied to a number of painted aluminum coupons. Table 1 provides contact angles for the resulting surfaces compared to those of the painted surface alone and a Fomblin Y oiled version of the painted surface. All of the commercial products increased wetting angles for water and ethylene glycol with an associated reduction in geometric surface energy. The coatings did not yield oleophobic characteristics; all surfaces were fully wetted by heptane. These treatments did not produce improved sliding or shedding characteristics for the painted surfaces. Also shown in Table 1 are values obtained for a paint only coupon that has been oiled with Fomblin Y. While this does not produce the same changes to contact angle as the commercial coatings, it does increase the water contact angle and that for heptane. In fact, this approach produces a 40° heptane contact angle, and heptane sheds from the oiled surface where it does not from the other materials. Given their intended use and the generally water shedding nature of automotive wax treatments, these result may appear to be inconsistent. In fact, on a reflective top coat, such as those used by the automotive industry, the results would be significantly different. The polyurethane paint system used here produces a flat finish and has no additional coating layers prior to application of the products. The reflective (shiny) surfaces that are generally achieved following application of automotive wax are not achieved on the coupons used here (Figure 1).

Table 1 – Sessile, Sliding, and Shedding Contact Angles

Coupon	Liquid	Sessile Angle	Sliding Angle	Shedding Angle	Geometric Surface Energy (mJ/m^2)
Painted Coupon	water	47.5 ± 1.1	>60	>60	71.9 ± 5.1
	ethylene glycol	55.7 ± 2.1	>60	>60	
	n-heptane	--	--	--	
Fomblin Y Oiled Paint	water	73.1 ± 2.1	>60	46.7 ± 3.3	32.2 ± 1.6
	ethylene glycol	52.5 ± 0.61	>60	49.8 ± 4.9	
	n-heptane	40.1 ± 2.9	>60	36.6 ± 3.3	
Nanoskin	water	95.9 ± 0.75	>60	>60	15.3 ± 0.86
	ethylene glycol	90.6 ± 1.5	>60	>60	
	n-heptane	--	--	--	
Wipe New	water	80.9 ± 0.89	>60	>60	27.2 ± 1.9
	ethylene glycol	77.9 ± 1.9	>60	>60	
	n-heptane	--	--	--	
NeverWet	water	107.9 ± 1.9	>60	>60	16.9 ± 1.8
	ethylene glycol	87.3 ± 0.95	>60	>60	
	n-heptane	--	--	--	
NanoWax	water	86.3 ± 1.9	>60	>60	21.2 ± 0.88
	ethylene glycol	72.7 ± 0.81	>60	>60	
	n-heptane	--	--	--	

The tendency of droplets to spread across the surfaces was also evaluated (Figure 2; Appendix A). For these studies, droplets of the simulants, DMMP, DFP, and MES (5 µL), were utilized. The spread of the droplets was quantified by measuring the diameter of the droplets in the images over time (Figure 3). For the paint only samples, MES and DFP spread quickly reaching the edges of the coupon at 10 and 2 min, respectively. DMMP does not spread during the course of the 30 min incubation for any surface except the NanoWax for which spreading is minimal. MES does not spread on the Nanoskin and Wipe New treatments and spreads very little on the NeverWet surface. The NanoWax treatment slows the spread of MES (Figure 2). Similarly to the painted coupon, the Nanoskin and NanoWax treatments allow DFP to spread and begin to evaporate during the 30 min incubation. The NeverWet surface significantly reduces the spread of DFP while the Wipe New surface reduces the spread even further (Figures 2 and 3).

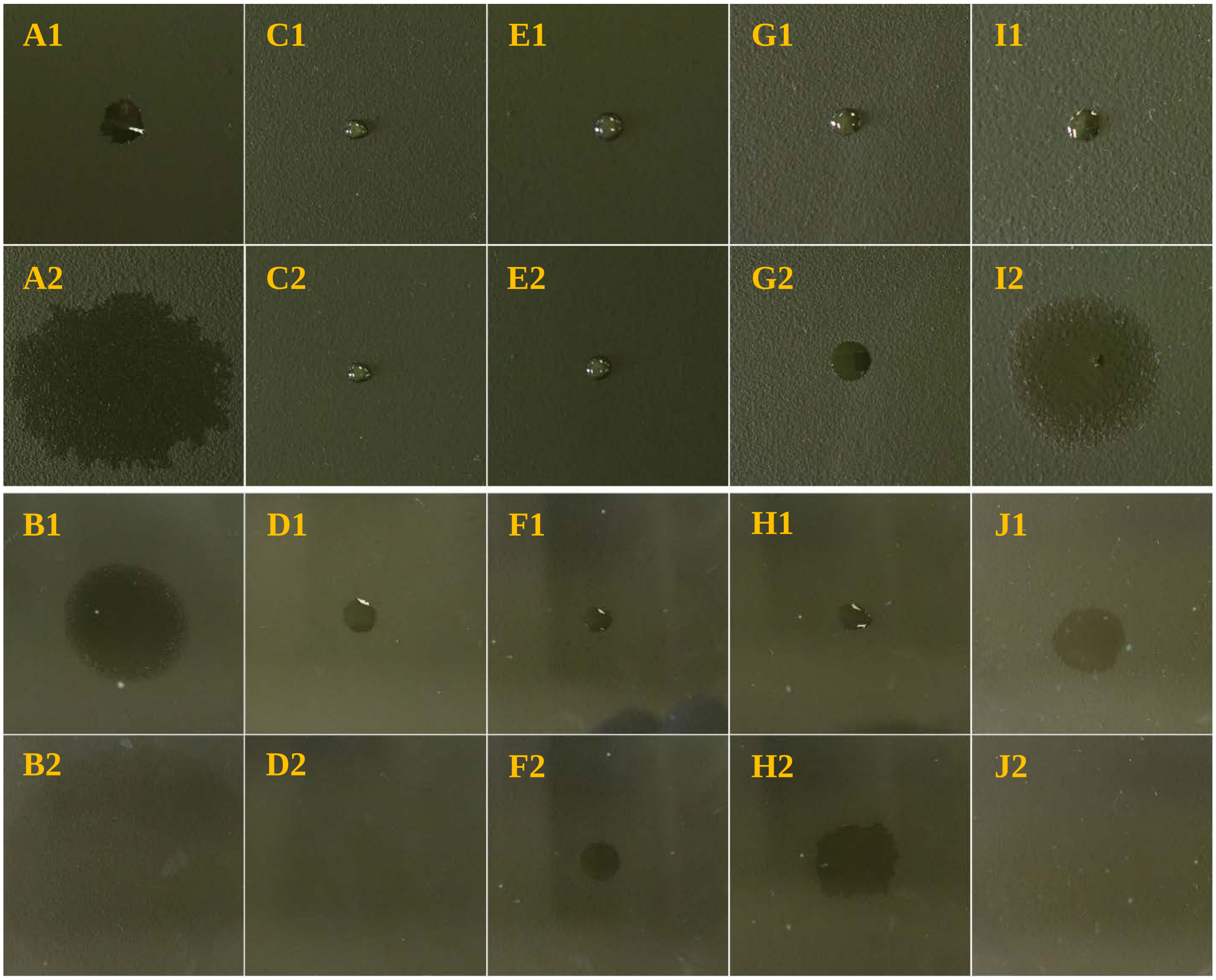

Fig. 2 — Images of painted coupons and painted coupons treated with the commercial products showing standing droplets of MES (A, C, E, G, I) and DFP (B, D, F, H, J) immediately following exposure (1) and after 30 min of aging (2): painted coupon (A and B), a Nanoskin treated coupon (C and D), a Wipe New treated coupon (E and F), a Never Wet treated coupon (G and H), and a NanoWax treated coupon (I and J).

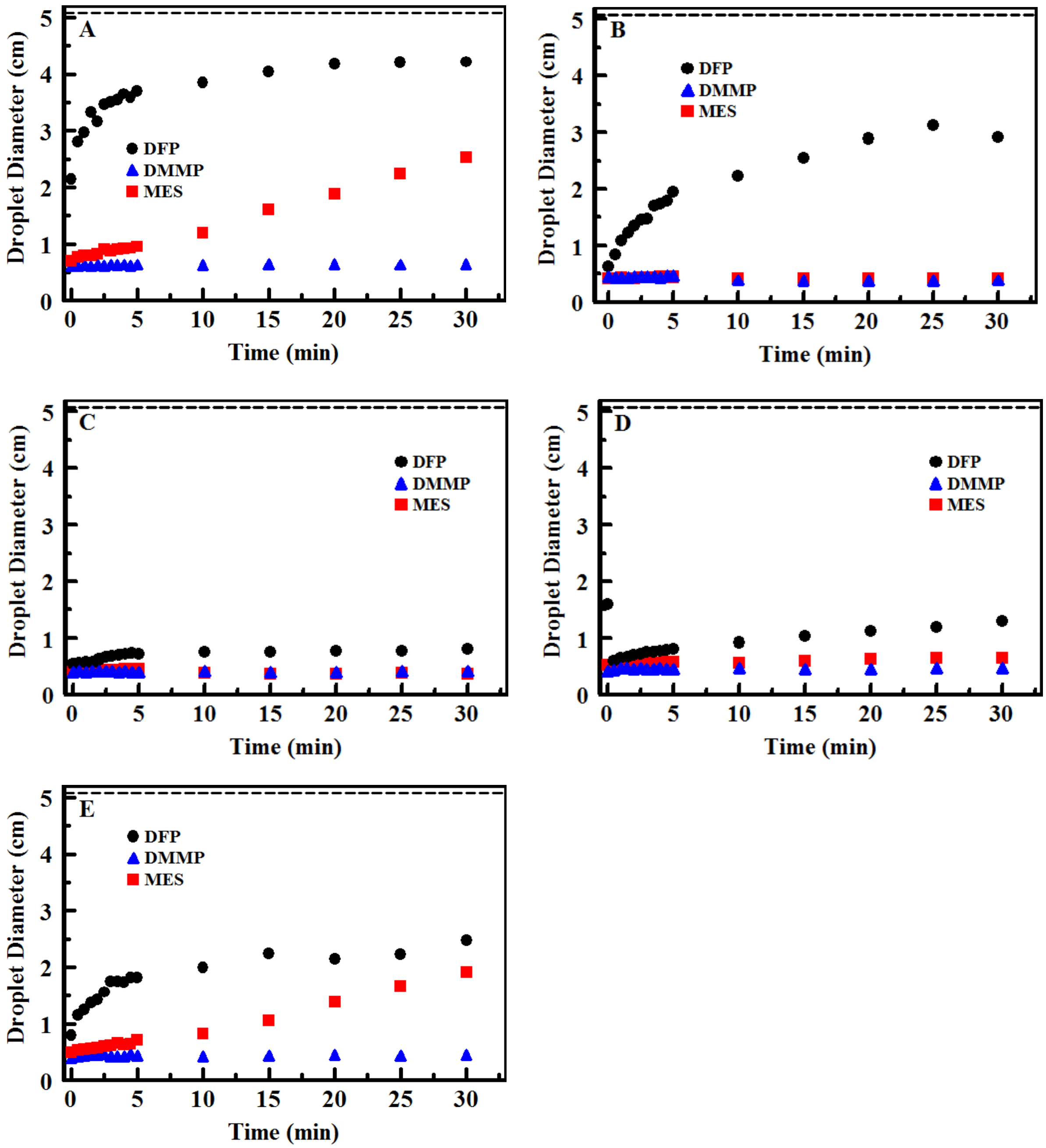

Fig. 3 — Progression of simulant droplet diameters during incubation on the surfaces for DFP (black), DMMP (blue), and MES (red): paint only (A), Nanoskin (B), Wipe New (C), NeverWet (D), and NanoWax (E).

The coupons were subjected to simulant exposure (5 g/m^2), aging, washing, and drying. The NanoWax, Nanoskin, and NeverWet coupons changed significantly upon extraction with isopropanol with wetting angles decreasing. These changes were not noted for the Wipe New treatment. When exposure was followed by the air and soapy water treatment for painted surfaces (Table 2), target retention was greater than 1 g/m^2 for paraoxon and MES. DMMP and DFP retention were significantly lower; DFP retention is

likely low due to evaporation of this target. Treated surfaces were subjected to exposure followed by the air and soapy water treatment, the DMMP retention by all of the treated surfaces was low (Table 2 and Figure 4). DFP retention was somewhat higher than DMMP with the Wipe New and NanoWax treatments retaining the lowest amounts. MES retention was higher still with the Wipe New treatment retaining the lowest levels. Paraoxon retention was the highest of the simulants with the NanoWax treatment retaining the least target.

For comparison purposes, paint only coupons that were not rinsed prior to isopropanol extraction retained the following: paraoxon – 4.90 g/m^2, MES – 4.81 g/m^2, DMMP – 4.95 g/m^2, DFP – 3.60 g/m^2. Though the nominal target application was 5 g/m^2, recovery from surfaces was always less than this value. Losses due to evaporation would be expected, especially for DFP. Additional losses likely occur during the rinse steps due to agent interaction with the untreated region of the coupon; the back of these coupons is unpainted aluminum.

Table 2 – Target Retention (g/m^2) Following 1 h Aging. A 5 g/m^2 challenge was used. ND indicates target concentrations below the threshold of detection for the method employed.

Coupon	Paraoxon	MES	DMMP	DFP
Painted Coupon	1.24	2.42	0.11	0.15
Fomblin Y Oiled Paint	0.75	1.83	0.03	0.01
Nanoskin	1.15	0.55	0.01	0.42
Wipe New	0.72	0.32	0.02	0.03
NeverWet	0.56	0.98	0.01	0.11
NanoWax	0.48	0.50	ND	0.08

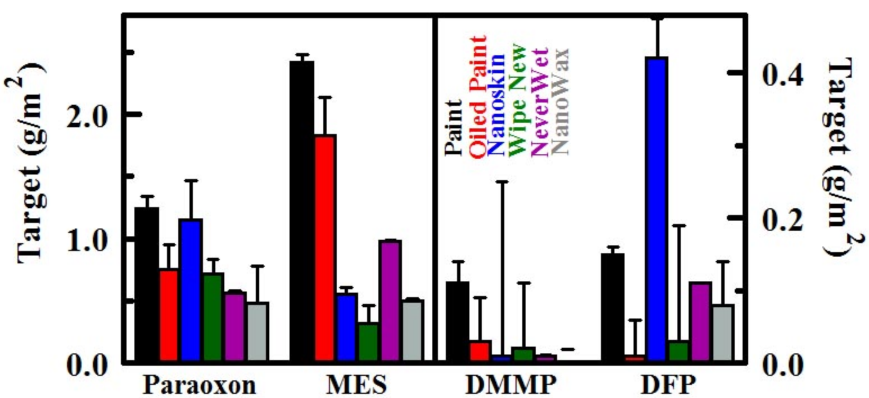

Fig. 4 — Target retention by coupons following treatment with an air stream and rinsing with soapy water: Fomblin Y oiled coupon (red); Nanoskin (blue); Wipe New (green); NeverWet (purple); and NanoWax (gray). Note the difference in scale between the paraoxon and DFP sides of the graph.

CONCLUSIONS

While the surface energy noted for the Wipe New treatment is higher than some of the better performing treatments evaluated under this effort, target retention by this treatment was similar to the most effective

materials evaluated. NanoWax also yielded low levels of retention. The commercial availability of these treatments means they would have all the advantages of COTS technologies. They are generally cheaper due to large quantity production, are more flexible for different types of applications, offer a shorter development to production cycle, and wide spread use will identify design defects sooner. Spectrophotometric analysis is necessary to determine the overall impact on color and reflectivity changes upon application of these treatments (Figure 2 and Appendix). The long term stability of the coatings should also be more thoroughly evaluated; however, their ease of application and relatively low cost should be noted.

ACKNOWLEDGEMENTS

The comments of Dr. Brent Mantooth (ECBC) on methods and agent analysis are appreciated. The authors would also like to thank Dr. Haewon Uhm (Rust-Oleum®) for her comments on the potential of Rust-Oleum® products. This research was sponsored by the Defense Threat Reduction Agency (DTRA, CB10125).

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Appendix

COUPON IMAGES

Fig. A1 — DFP on paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1.0 (D), 1.5 (E), 2.0 (F), 2.5 (G), 3.0 (H), 3.5 (I), 4.0 (J), 4.5 (K), 10 (L), 15 (M), 20 (N), 25 (O), and 30 (P) min following application of the target. These images were collected with a glass cover in place to limit evaporation. Reflections from the cover can be seen in some images.

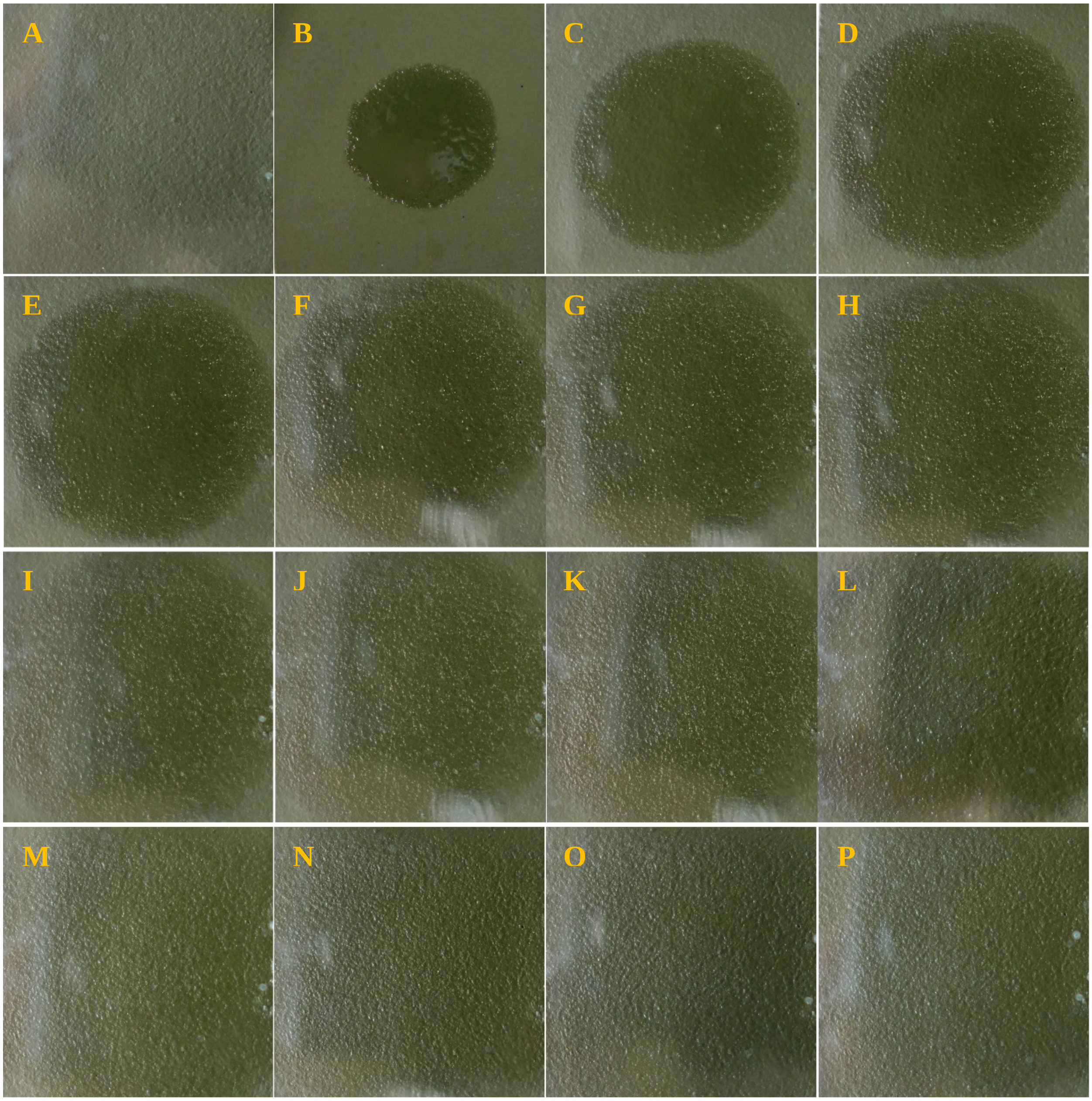

Fig. A2 — MES on paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

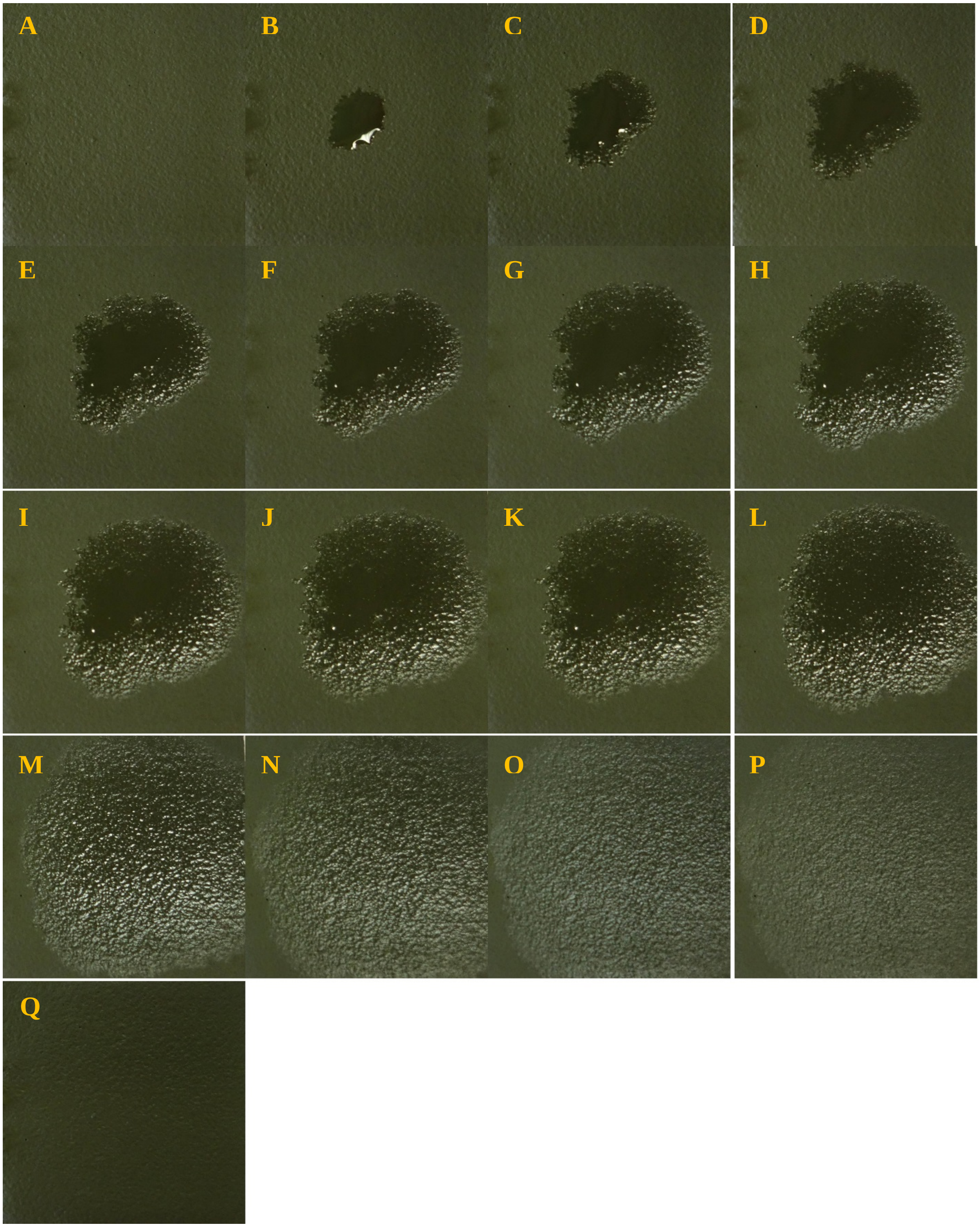

Fig. A3 — DMMP on paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

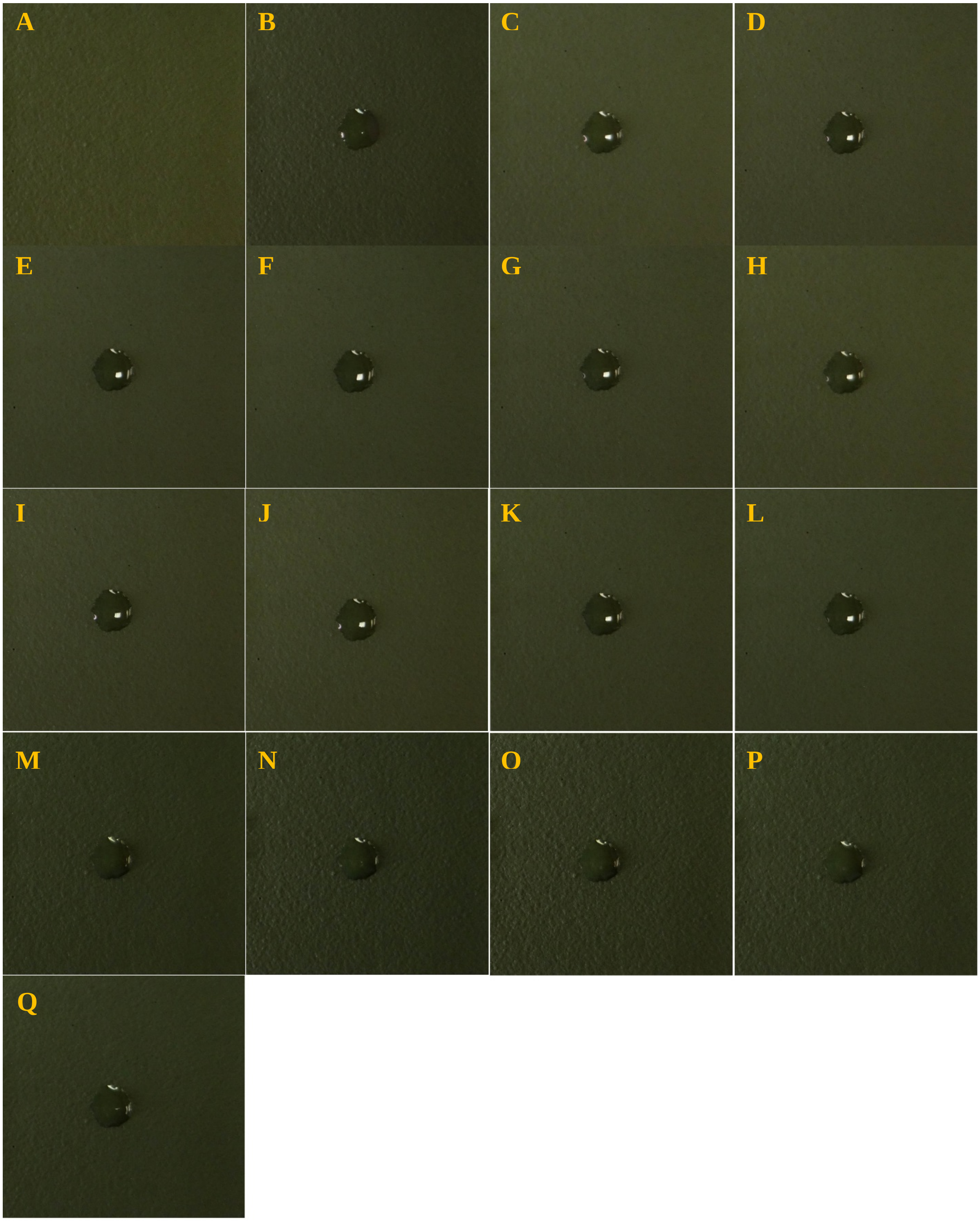

Fig. A4 — DFP on Nanoskin. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target. These images were collected with a glass cover in place to limit evaporation. Reflections from the cover can be seen in some images.

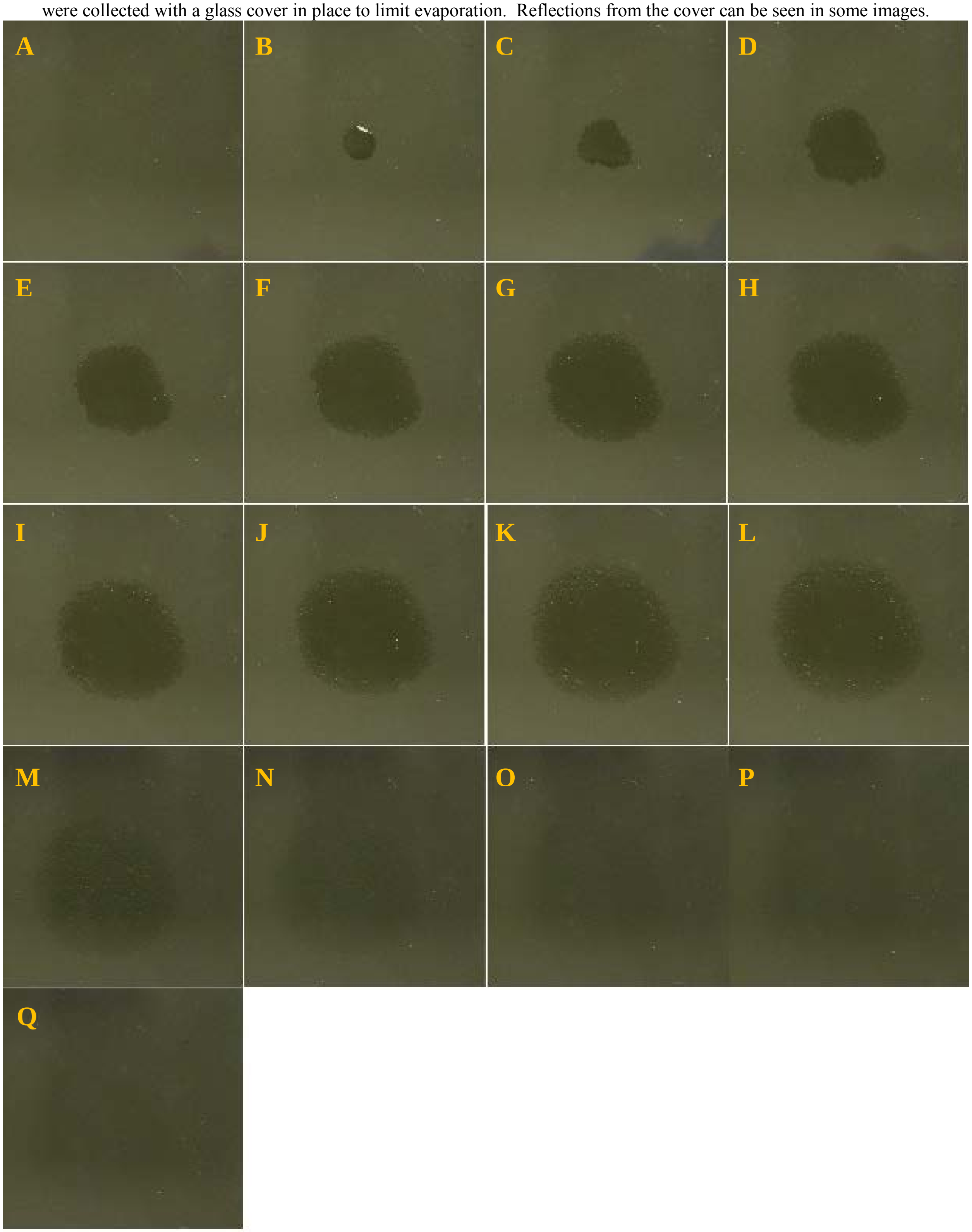

Fig. A5 — MES on Nanoskin. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

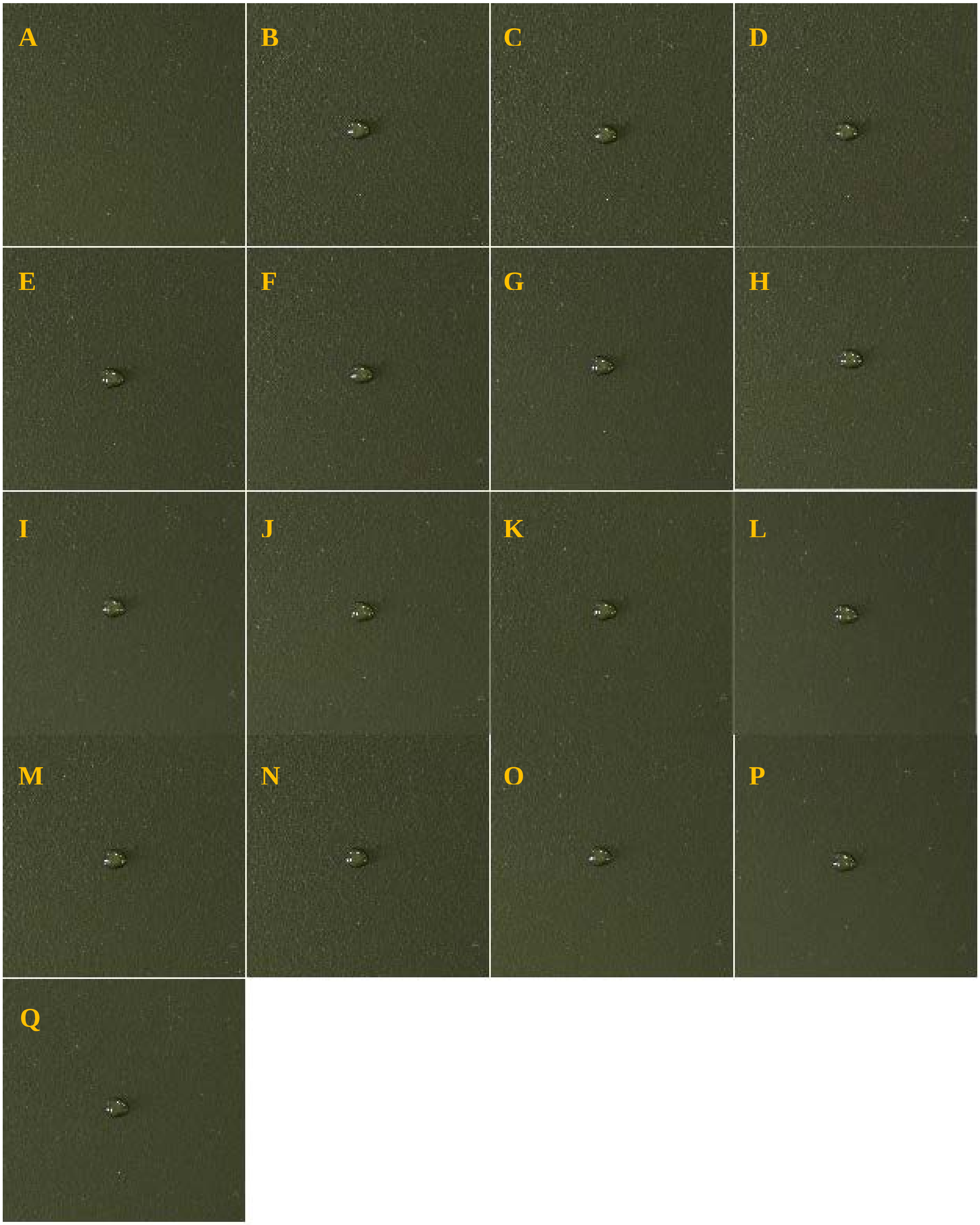

Fig. A6 — DMMP on Nanoskin. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 5.5 (M), 10 (N), 15 (O), 20 (P), 25 (Q), and 30 (R) min following application of the target.

Fig. A7 — DFP on Wipe New. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target. These images were collected with a glass cover in place to limit evaporation; reflections from the cover can be seen in some images.

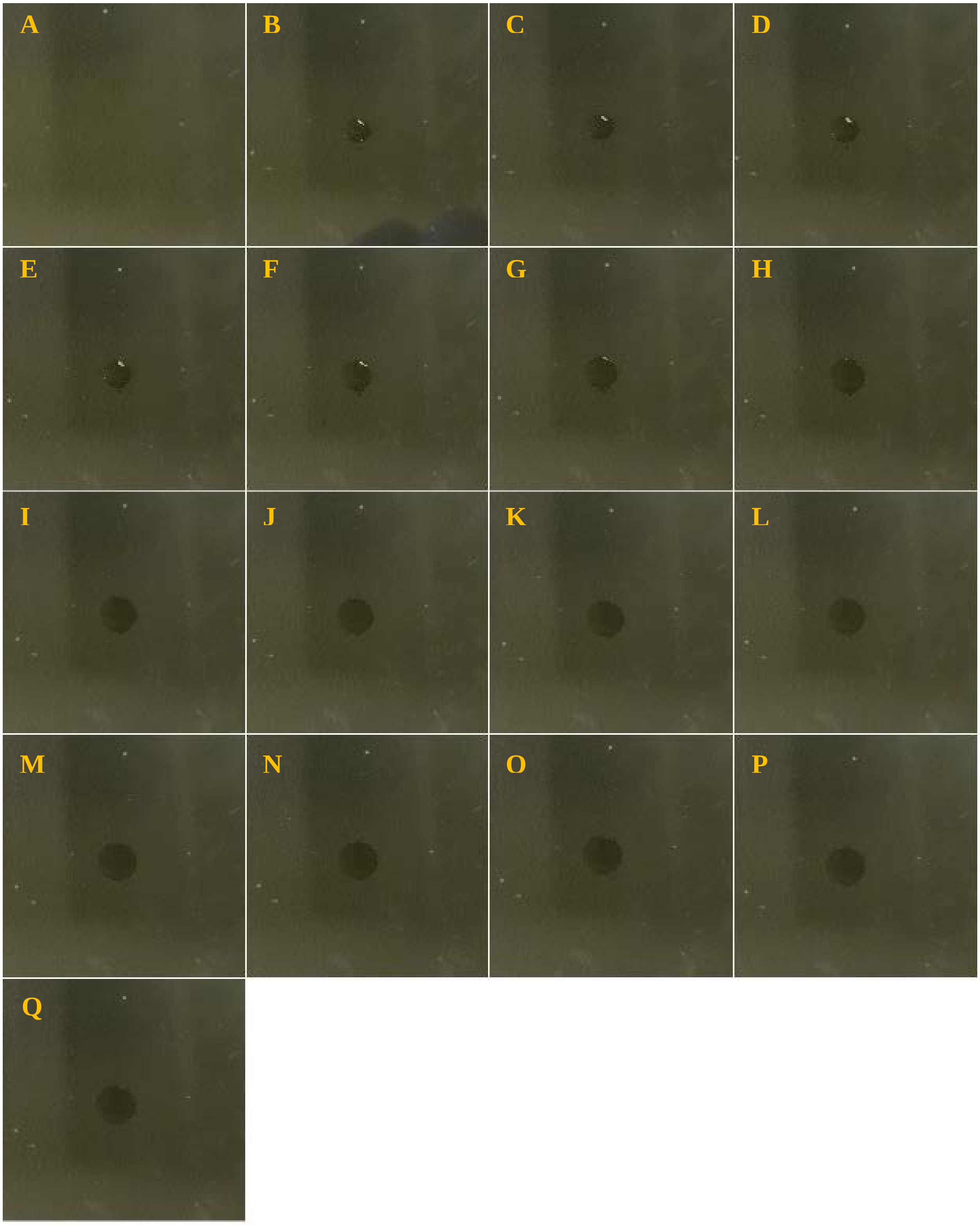

Fig. A8 — MES on Wipe New. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

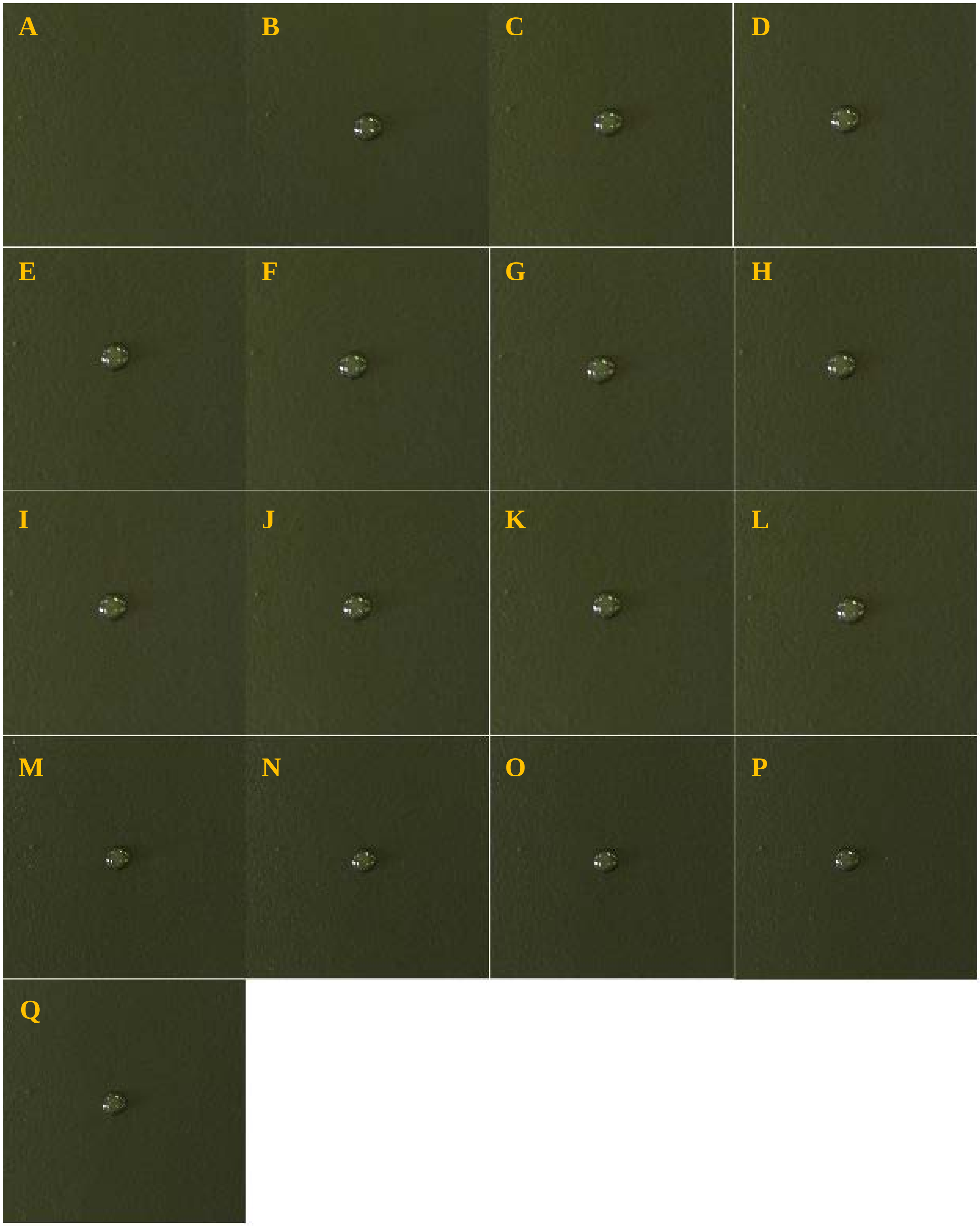

Fig. A9 — DMMP on Wipe New. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

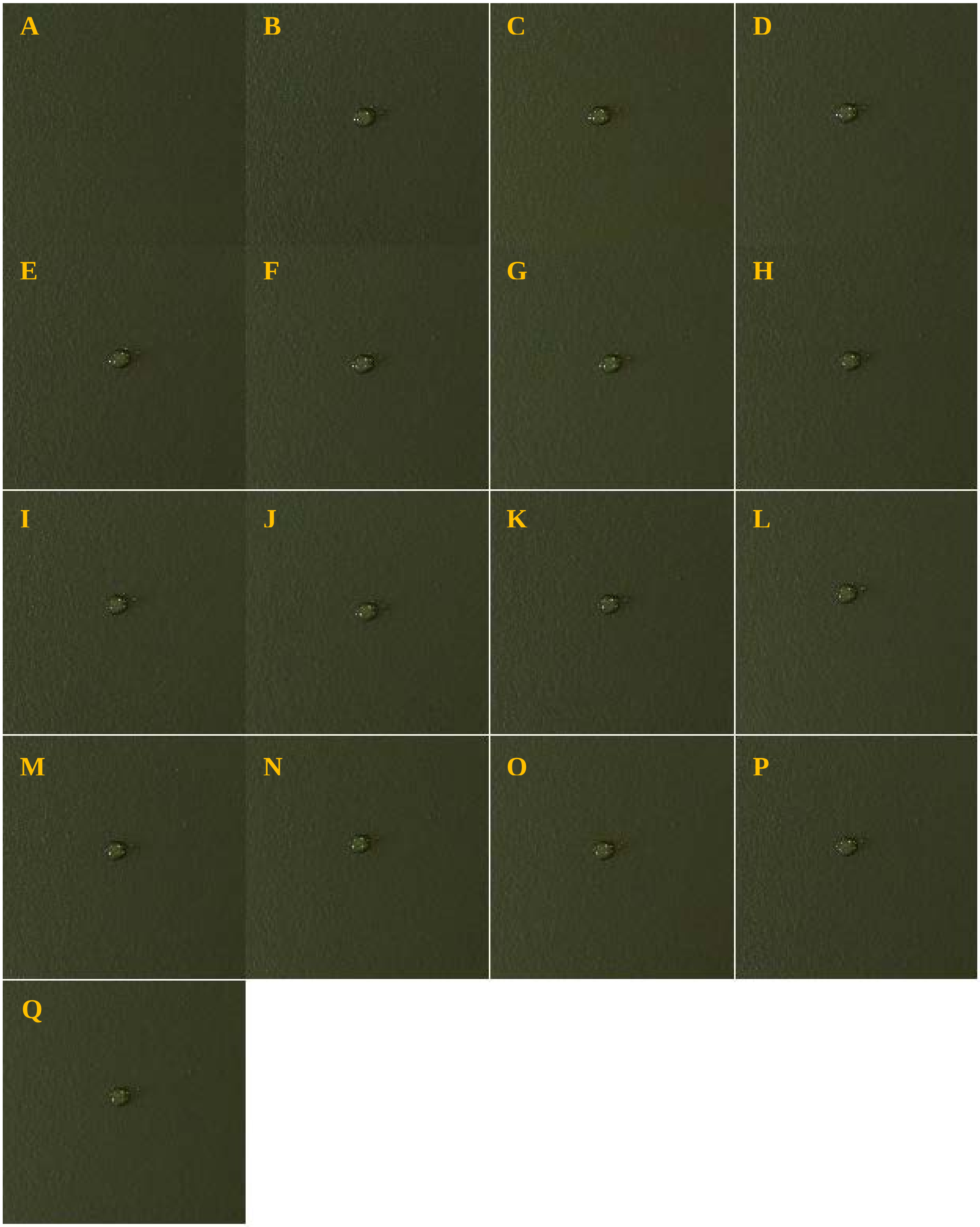

Fig. A10 — DFP on Never Wet. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target. These images were collected with a glass cover in place to limit evaporation; reflections from the cover can be seen in some images.

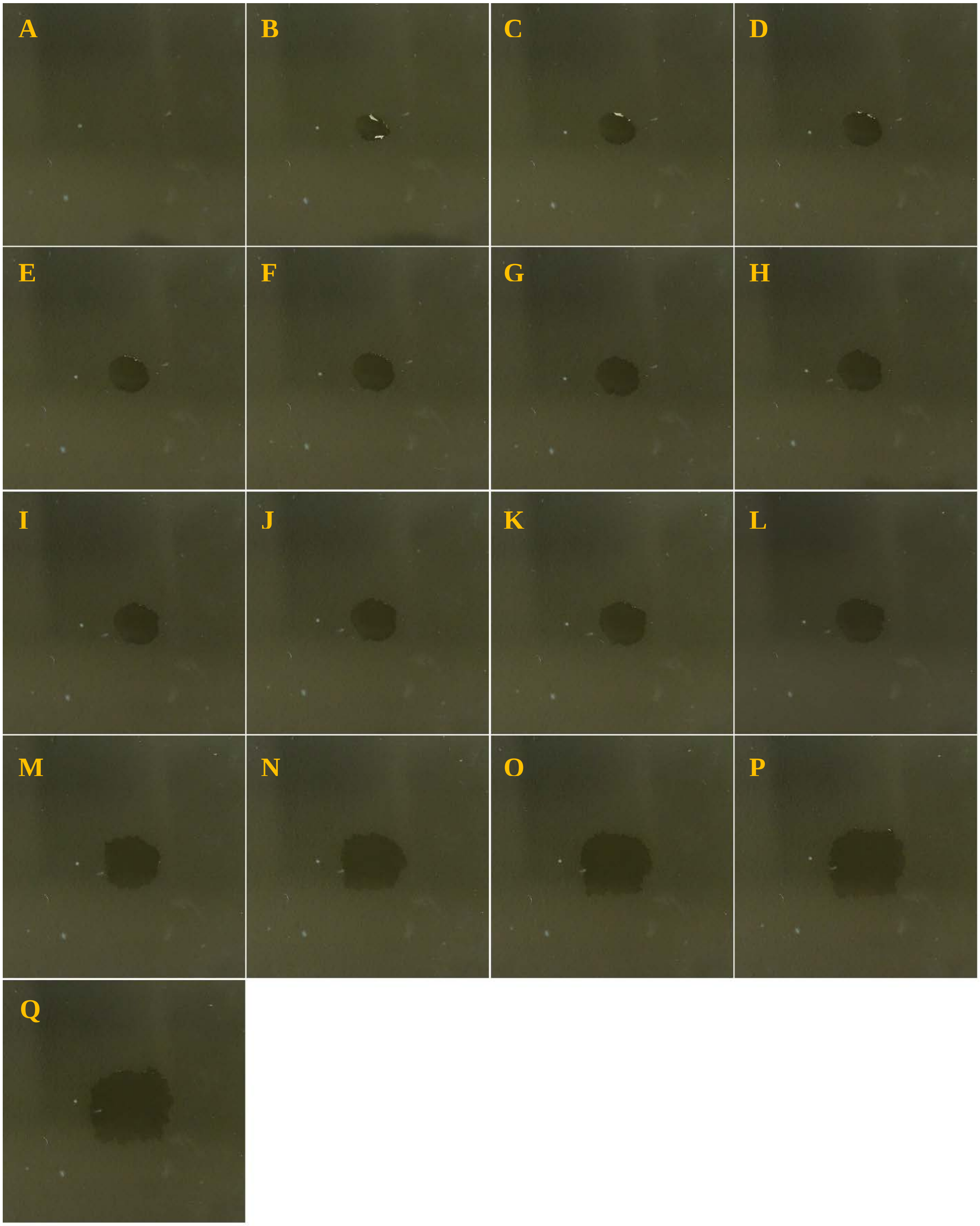

Fig. A11 — MES on NeverWet. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

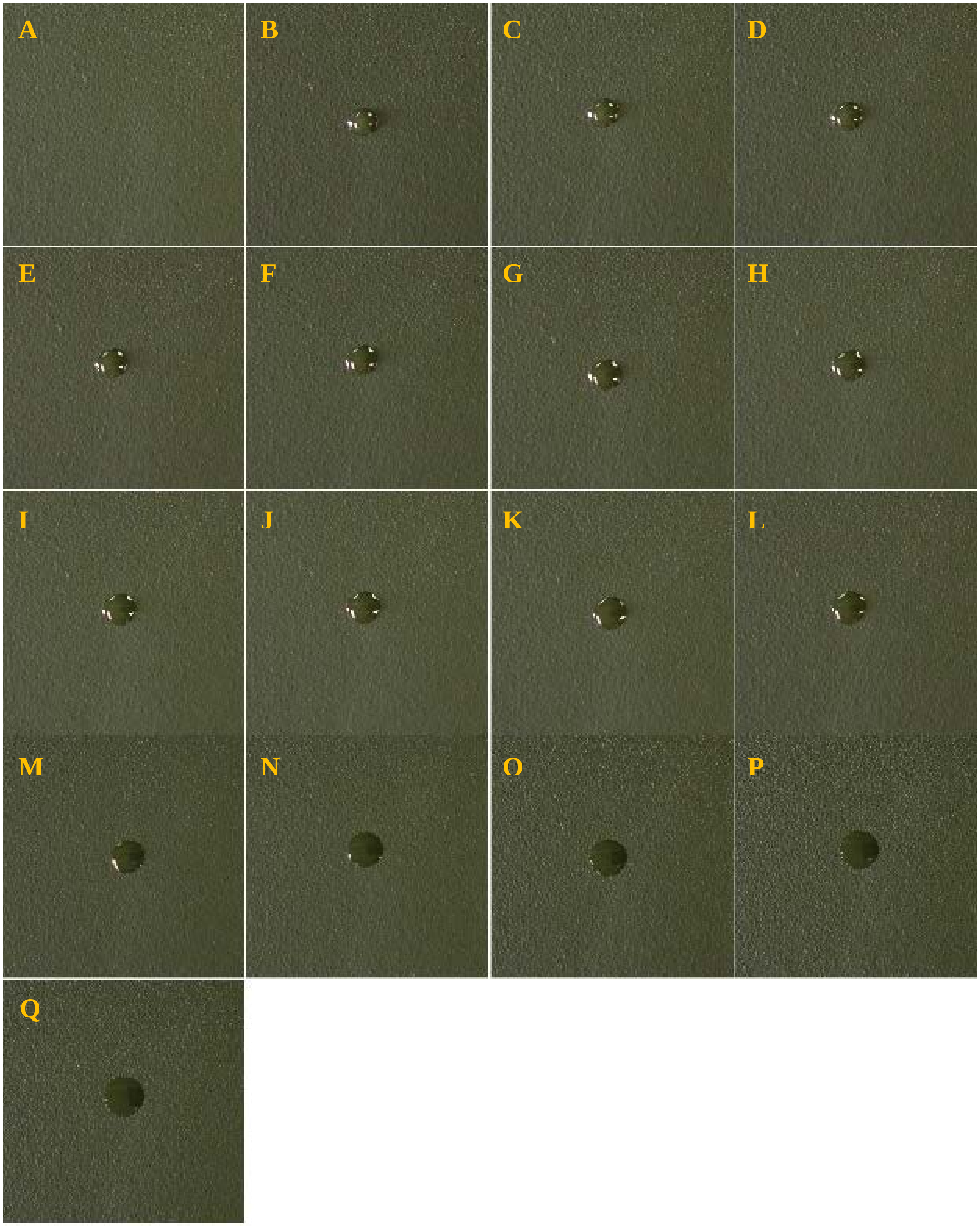

Fig. A12 — DMMP on NeverWet. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

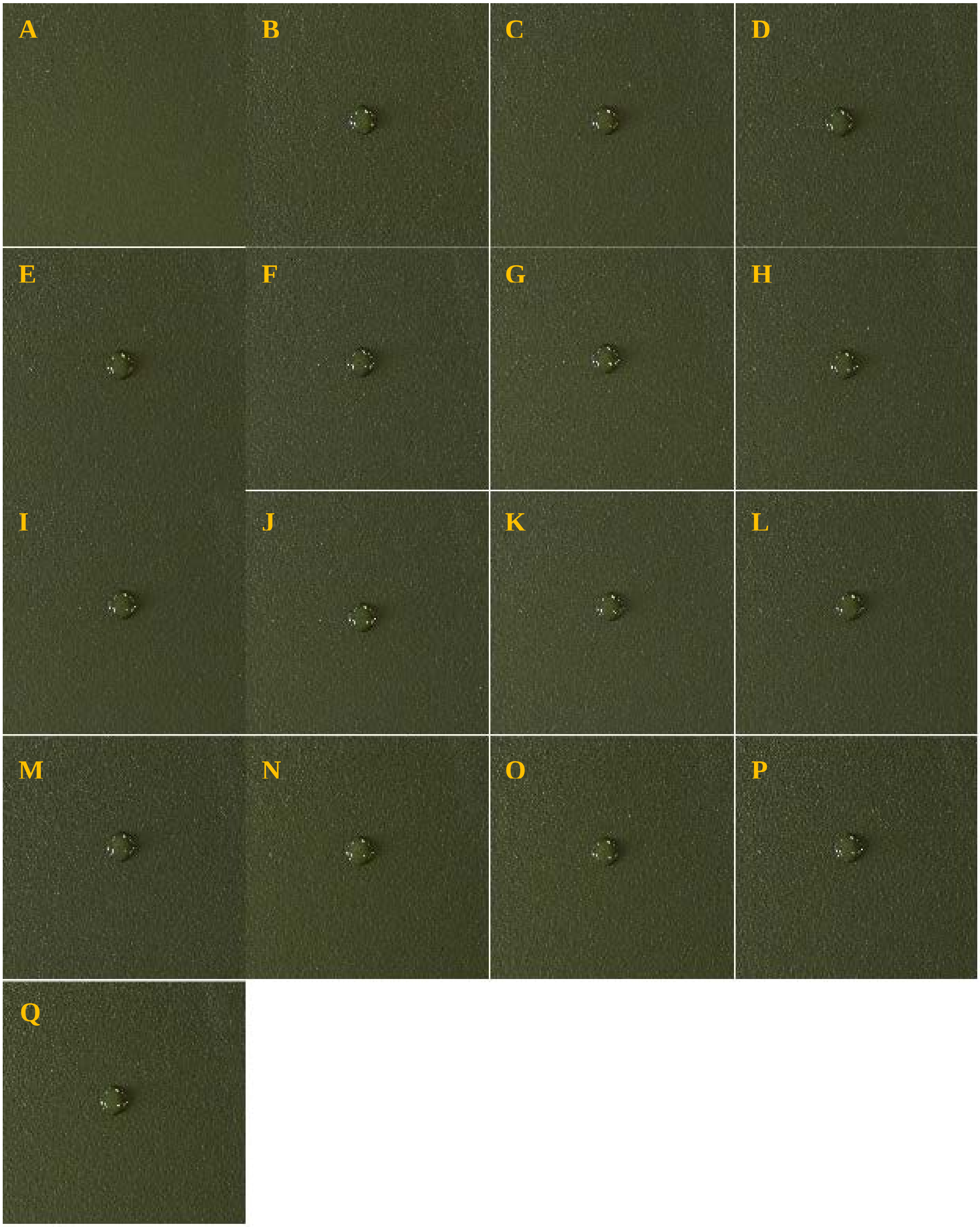

Fig. A13 — DFP on NanoWax. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target. These images were collected with a glass cover in place to limit evaporation; reflections from the cover can be seen in some images.

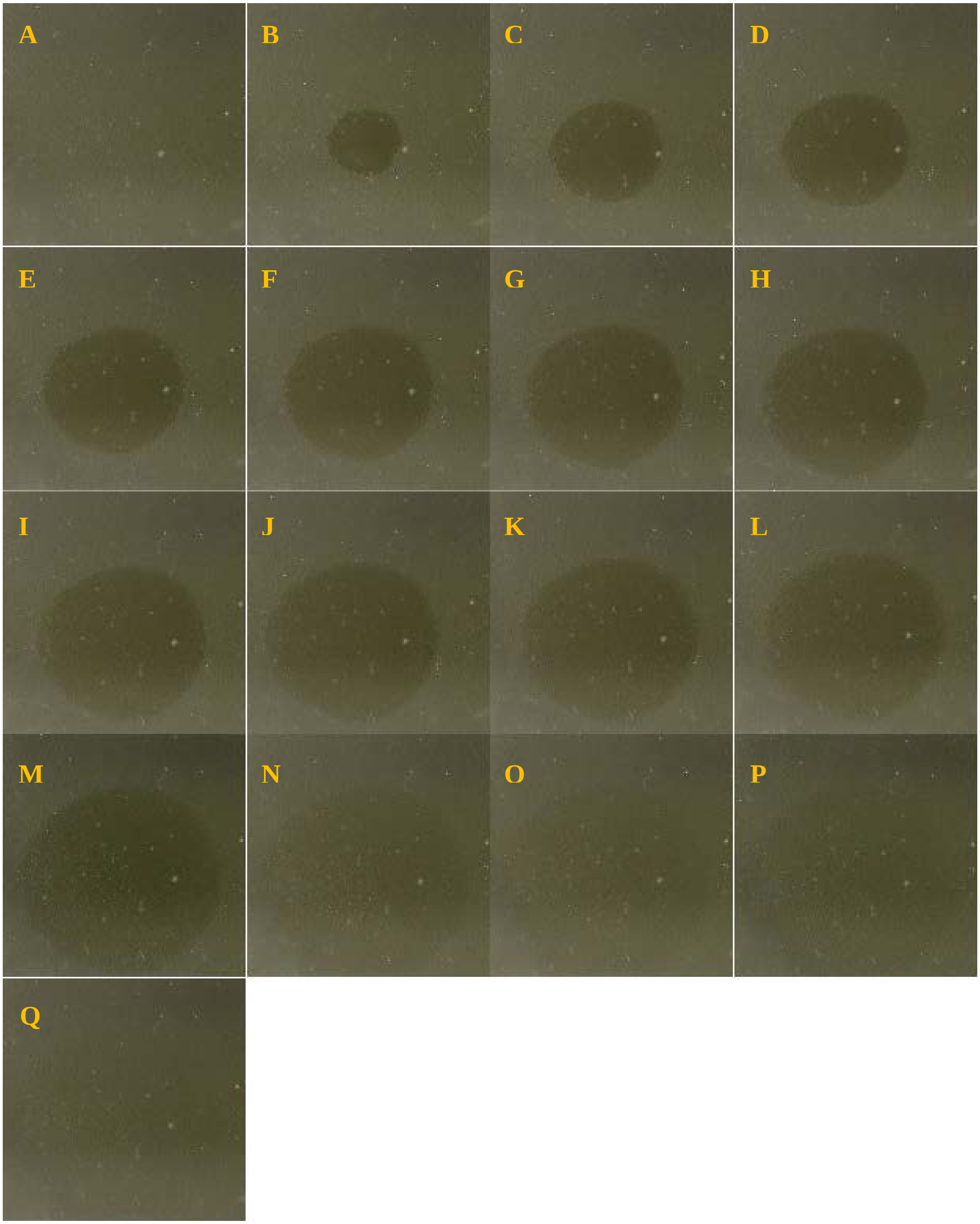

Fig. A14 — MES on NanoWax. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

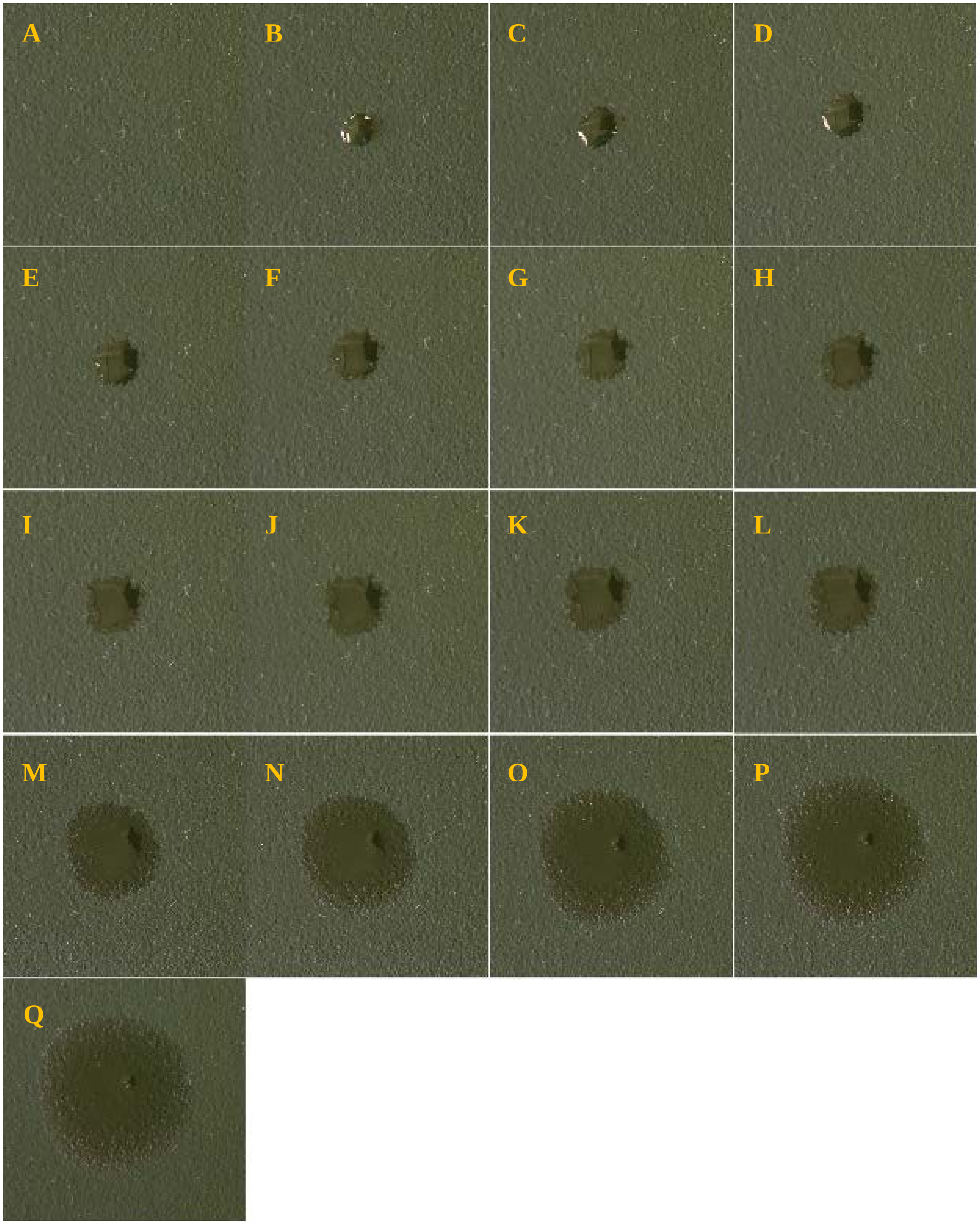

Fig. A15 — DMMP on NanoWax. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.